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IS 1448-134 (1989): Methods of and Test for Petroleum its Products, Part 134: Determination of Volatile Matter in Raw and Calcined Petroleum Coke [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS 1448 (P : 134) : 1989
Adopted 5 April 1989
(Reaffirmed 2002)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 134]

DETERMINATION OF VOLATILE MATTER IN RAW AND CALCINED PETROLEUM COKE

(Second Reprint MAY 1999)

UDC 665 777·4 : 543·813

Adapted with permission from ASTM D4421-84

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1 SCOPE

1.1 This standard covers a test method for determination of volatile matter produced by pyrolysis or evolved when petroleum coke, or both, are subjected to specific conditions of the test.

1.2 This test method is empirical and requires the entire test procedure to be closely followed to ensure results from different laboratories to be comparable.

2 REFERENCE

2.1 The Indian Standard 'IS 1447 (Part 4) : 1989 Sampling of petroleum coke for laboratory analysis' is a necessary adjunct to this standard.

3 OUTLINE OF THE METHOD

3.1 Volatile matter of a moisture free petroleum coke is determined by measuring the mass loss of the coke when heated under the exact conditions of this test procedure.

4 SIGNIFICANCE AND USE

4.1 The volatile matter of petroleum coke affects the density of coke particles and may affect artifacts produced from further processing of the coke.

4.2 The volatile matter may be used in estimating the calorific value of coke.

5 INTERFERENCES

5.1 Moisture has a double effect. The mass loss is increased and the moisture free sample mass is decreased by the amount of moisture actually present in the test sample.

5.2 Particle Size Effect

5.2.1 For any given coke there is a relationship between the sizes of the original particles in the sample and the volatile matter of the particles. The larger the particle size, the lower the volatile matter. For this reason, representative sampling and division of the sample are very important.

5.2.2 The particle size range of the analysis sample affects the volatile matter. The coarser the analysis sample, the lower the volatile matter will be. The analysis sample is crushed to pass 250 micron IS sieve but is not overcrushed. A No. 125 IS sieve should retain 40 to 55 percent of the sample. The analysis sample is not to be obtained by scalping and discarding a portion of the sample.

5.2.3 Any segregation of particle sizes within the analysis sample should be corrected by reblending the sample just prior to weighing the test sample.

5.3 Downward drift of furnace temperature caused by an increase in the millivolts per degrees Celsius generated by an aging type K thermocouple produces a lower volatile matter value.

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5.4 Diffusion of air into the crucible caused by a poor fit between the crucible and its cover or by any other source of available oxygen causes high and earratic volatile matter values.

5.5 Free or tramp iron or mill scale in the coke coats the surface of the platinum crucible and decreases its life. The coating tends to form an oxide film during the preburning step (*see* **10.1**). The oxide film provides a source of excess mass loss equal to almost twice the oxygen mass gained. Clean the crucible and buff with sand. Buffing will restore lustre to the surface of the crucible.

6 APPARATUS

6.1 Vertical Tube Furnace, Fieldner, Electric, calibrated and regulated to maintain a temperature of 950 ± 15°C as measured by a thermocouple mounted inside the furnace.

6.2 Nickel Chromium Crucible Support, for the platinum crucible.

6.3 Platinum 90 Percent — Rhodium 10 Percent — Crucible, volatile matter form, reinforced top and bottom, capacity 15 ml, height 33 mm, top diameter 29 mm, fitted with a capsule type cover 10 mm high. The cover is to have a 0·5 mm diameter hole drilled through the centre. The total mass of crucible and cover, approximately 20 g.

6.4 Reshapers, for the platinum crucible and cover.

6.5 Platinum Tipped Stainless Steel Tong, 200 mm long.

6.6 Sea Sand, rounded particles about 50 mesh.

6.7 Crucible, porcelain high form 30-ml capacity.

6.8 Timers, stop watch or second-timer accurate to ± 1 second.

7 PRECAUTIONS

7.1 Effusion of gaseous products including soot and various hydrocarbons and the increase of heat associated with the test, may make use of a hood desirable.

8 PREPARATION OF FURNACE

8.1 If desired, the radiant heat loss to the room from the outside metal cylinder may be reduced by insulating the furnace with a thick layer of insulation and glass wool.

8.2 Fit thermocouple into the bottom of the furnace. Use borosilicate glass wool to close the opening around the thermocouple. Adjust the thermocouple position so that it rests 2 to 3 mm below the bottom of the platinum crucible with the platinum crucible positioned in the crucible support and the support resting on the top of the furnace.

8.3 A 100 to 150 mm mirror should be hung above the furnace, positioned to allow the operator to observe the platinum crucible during the time it is positioned in the furnace.

8.4 A set up for this test for manual operation is shown in Fig. 1.

9 HANDLING AND CLEANING OF PLATINUM CRUCIBLE

9.1 Handling the Platinum Crucible

Always handle the platinum crucible and its cover gently, using platinum clad stainless steel crucible tongs; avoid handling with the finger. Place the platinum crucible with its cover in a porcelain crucible (30 ml capacity) which serves as a 'carrier' and protects it from coming in frequent contact with the tongs, thereby increasing the life of the platinum crucible. When not in use, the platinum crucible in its carrier should be stored in a desiccator. Do not handle the carrier with the platinum clad tongs.

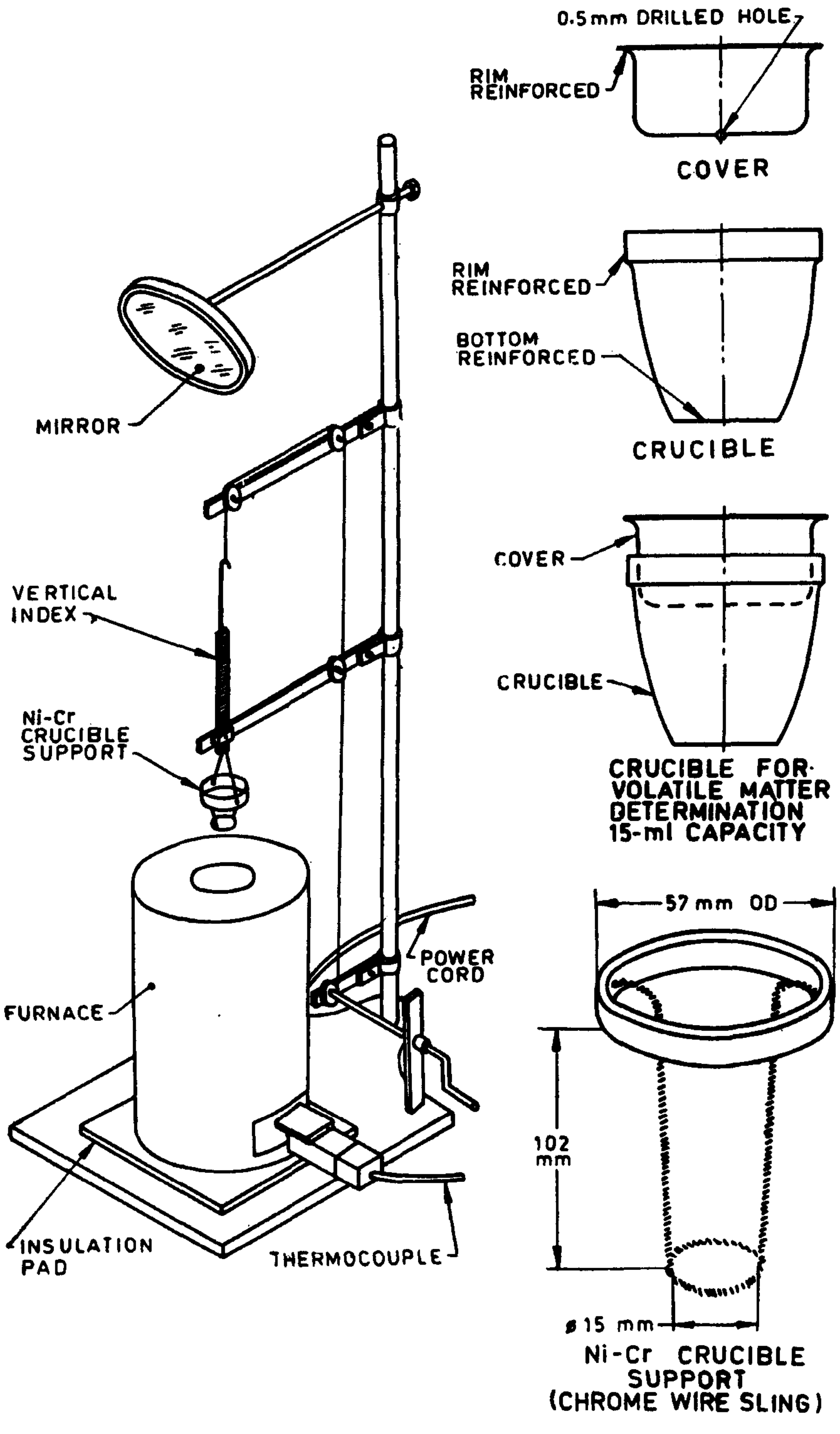
9.2 *Cleaning and Care of the Platinum Crucible*

Depending on the frequency of its use, the crucible should be periodically cleaned with potassium pyrosulphate; this is done by adding about 1 g of the pyrosulphate; to the crucible and fusing carefully, followed by rinsing with hot water, polishing with sea sand and drying. A mirror like smooth surface on the platinum should be maintained by frequent polishing with sea sand. Any dents or wrinkles on the crucible should be immediately smoothed out using re-shaper. All unnecessary handling and re-forming is to be avoided.

10 PROCEDURE FOR RAW PETROLEUM COKE

10.1 Ignite a platinum crucible and cover at 950°C for 5 minutes periods to constant mass (± 0·5 mg). A burner or any furnace may be used but the crucible should be set in a nichrome wire support regardless of the style of furnace used. The cover should not seal the crucible at this time so that the carbon will burn off. Cool to room temperature in a desiccator before weighing the crucible and cover. Do not keep the crucible at room temperature more than 20 minutes before it is weighed. Record all masses to 0·1 mg.

10.2 Avoiding segregation of particles, transfer 1 g (± 1 mg) of the dry analysis sample [*see* **5.4.3.6** and **5.4.4.6** of IS 1447 (Part 4) : 1989] to the



The determination of volatile matter.

FIG. 1 SET-UP FOR THE DETERMINATION OF VOLATILE MATTER

crucible and weigh the crucible with its content and cover to the nearest 0·1 mg. Fit the cover on the crucible and press it gently into position. Avoid physical distortion of the crucible and cover.

10.3 Set the crucible vertically in the holder and centre it over the furnace opening with the bottom of the crucible in line with the top surface of the transite furnace cover (*see* Note). Be sure neither the crucible nor the holder touch the wall of the furnace. Start the timer.

NOTE — There are some coke volatile matter relationships which require the crucible bottom to be 6 mm above the top surface of the transite cover in the initial position. These relationships prevent control of sparks or flames or both.

10.4 After 60 s (± 5 s), lower the holder approximately 8·5 mm into the furnace. The holder is lowered another 8·5 mm after each total elapsed time of 120, 180, 240, 300 and 360 s (each ± 5 s). After the move at 360 s the top of the crucible should be 19 mm below the top surface of the transite cover. Do not position the bottom of the crucible more than 57 mm below the top surface of the transite cover at this time (*see* Note).

NOTE — The heating rate is extremely important since the test is empirical and equilibrium is not achieved. The heating rate is controlled by the rate of lowering the crucible into the furnace. Once the holder is positioned immediately over the furnace, the time schedule shall be observed. The crucible should not be removed, or raised and re-lowered, without verifying the test.

10.5 If at any time during the test, sparking occurs such that the sparks are seen to be above the height of the crucible cover or if a flame or flames occur such that the sum of all visible simultaneous flames would extend above the height of the crucible cover, the test shall be repeated with slight variations during the first 600 s. The variations are to be made with respect to the position when the sparks or flames occur. A more severe heat up rate (more rapidly lowering) will result in less sparking or flaming subsequently but may result in sparking or flaming earlier in the test. Discretion is necessary. A maximum furnace depth for the bottom of the crucible of 57 mm is to be maintained during the first 600 s.

10.6 After 600 s (± 5 s). total elapsed time, lower the holder so that the brass ring of the holder rests directly upon the transite furnace cover (maximum depth in furnace).

10.7 After 960 s (± 5 s), total elapsed time, raise the holder and crucible out of the furnace and immediately place the crucible in a desiccator.

10.8 Weigh the crucible after it has cooled to room temperature and before it has been at room temperature for longer than 20 minutes.

11 PROCEDURE FOR CALCINED PETROLEUM COKE

11.1 Weigh into the clean platinum crucible 3 g (± 1 mg) of the dry analysis sample passing through 250 micron IS sieve [*see* **5.4.3.6** and **5.4.4.6** of IS 1447 (Part 4) : 1989]. Fit the capsule cover on the crucible and press it gently into position. Place the loaded crucible into the wire sling.

11.2 Lower the crucible into the furnace at its maximum depth so that the brass ring of the holder rests directly upon the transite furnace cover. Start the stop watch.

11.3 Allow to remain in this position exactly for 7 minutes. Then, rapidly remove the crucible from the furnace. Allow to cool in a desiccator to room temperature and weigh to the nearest 0·1 mg.

11.4 Replace the crucible inside the furnace, in a manner similar to above and heat the crucible for a further 7 minutes. Cool and weigh again to the nearest 0·1 mg. The loss of mass during second heat treatment is attributed to loss due to oxidation of coke.

11.5 Deduct this mass loss due to oxidation from the mass loss recorded after the first heating to obtain the actual mass loss of the sample.

12 CALCULATIONS

12.1 Calculate the volatile matter of the moisture free prepared coke as follows:

$$\text{Volatile matter, percent by mass} = (A/B) \times 100$$

where

A = mass loss, in g, of the sample; and

B = mass, in g, of the sample.

13 REPORT

13.1 For Raw Petroleum Coke

The results should be the average of duplicates and should be reported to the first decimal place.

13.2 For Calcined Petroleum Coke

The results should be the average of duplicates and should be reported to include two significant figures.

14 PRECISION

14.1 Results of the duplicate tests shall not differ by more than the following limits:

	Repeatibility	*Reproducibility*
For raw petroleum coke	0·2	0·5
For calcined petroleum coke	0·1	0·3

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